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Research Article

Low-Temperature Synthesis of Vanadium Dioxide Thin Films by Sol-Gel Dip Coating Method

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The vanadium dioxide (VO_2) thin films were synthesized by sol-gel dipping on a glass slide substrate at low temperature of 500°C in a vacuum tube furnace at a pressure of 2×10^{-3} mbar by 2-step calcination without an intermediate gas purging. Synthesis conditions, including temperature, vacuum pressure, and calcination steps in the vacuum tube furnace, were investigated to find the optimum condition that promoted the formation of VO_2 phase. It was found that the 2nd calcination step was very important in realizing the monoclinic vanadium dioxide (VO_2 (M)). The results of the valence electron analysis revealed the outstanding phase of VO_2 and a small amount of V_2O_5 and V_2O_3 phases. The small crystallites of the VO_2 were homogeneously distributed on the surface, and the grain was of an irregular shape of ~220–380 nm in size. The film's thickness was in a range of 69–74 nm. The film exhibited a metal-to-insulator transformation temperature of ~68°C and good thermochromic property. Visible optical transmittance remained at ~40–50% when the sample's temperature changed from 25 to 80°C for a near infrared (NIR) region.

1. Introduction

Vanadium oxide presents a partly-filled 3d orbital, leading to the formation of several oxides such as VO, V_2O_3, V_4O_7, VO_2, V_2O_5, and V_6O_{13}, which all belong to the series of Magneli (V_nO_{2n-1}) and Wadsley ($V_{2n}O_{5n-2}$) systems [1–3]. Among these oxides, monoclinic vanadium dioxide (VO_2 (M)) has gained most attention owing to its fascinating thermochromic property that can switch a phase structure reversibly between a monoclinic (semiconductor) and a rutile (metal) phase at about 68°C [3, 4] in response to external stimuli, leading to dramatic changes in the electrical and optical properties. The change of optical property from infrared-transmitting at low temperature to infrared-reflecting at temperatures higher than the monoclinic-rutile phase transition temperature, while maintaining the visible transmittance, makes the vanadium dioxide a promising candidate as thermochromic coating for a variety of devices such as smart windows, tunable absorbers and emitters, and thermal rectification [4, 5].

The thermochromic VO_2 (M) thin films have been prepared by several methods. The most typical methods include physical vapor deposition, chemical vapor deposition, and sol-gel method. The physical and chemical vapor depositions can precisely control the stoichiometry of films by monitoring oxygen partial pressure. However, the complex deposition processes and expensive equipment significantly limit their practical applications [6, 7]. The sol-gel method has been widely employed for material investigation because of several advantages. For instance, it can be coated on complex shape, large substrate. The process is of low cost, and there is easy control of reaction kinetics and atomic doping. In addition, lower calcination temperature is typically required to obtain crystalline structure [8–10]. In a typical sol-gel process for VO_2 preparation, a V^{5+}-containing precursor is firstly prepared, and then the precursor is deposited onto a clean, hydrophilic substrate through dip or spin coating. The obtained sample is dried and calcined typically at ≥600°C [11–13] to transform an amorphous phase to a desired crystalline structure. Meanwhile, part of V^{5+} ions was reduced to V^{4+} ions [8, 11].

For commercial applications, such as smart window coating, glass is the desirable substrate. The soda lime glass is the most common and cheapest one [14, 15]. Considering that, for wet-chemical processes, the crystalline VO_2 phase is typically formed by a high-temperature treatment process. This would restrict the calcination of VO_2 thin films on the soda lime glass substrate which is generally tempered at approximately 600°C. The glass begins to soften at this temperature. The strength of the tempered glass would be weakened after VO_2 thin film deposition at temperatures higher than its softening temperature. Therefore, for soda lime glass panel substrates, low-temperature deposition (<600°C) is of significant importance for the VO_2 thin film preparation under inert atmosphere, e.g., vacuum or $N_2/Ar/H_2/CO/CO_2$ [7, 8, 10, 12, 14, 16, 17]. Moreover, most of the methods reported in literatures employed low vacuum pressure of tube furnace, which utilizes high power of the turbo and diffusion vacuum pumps [10, 12, 14, 17]. In some cases, intermediate gases were purged in the tube furnace, during calcination, to prevent film's surface from oxidation in the atmosphere [7, 8, 12, 14, 16, 17].

In this work, the outstanding VO_2 (M) phase was prepared via sol-gel dip coating on a soda lime glass slide at low calcination temperature of 500°C by using only a turbo pump to generate low vacuum pressure of 2×10^{-3} mbar in a tube furnace. Moreover, no intermediate gas was purged during a reducing step. Synthesis conditions, which include vacuum pressure, calcination temperature, and steps in the vacuum tube furnace, were investigated. Electrical resistance and optical properties of the prepared vanadium dioxide film were characterized.

2. Materials and Methods

2.1. Synthesis of Vanadium Oxide Thin Films. The vanadium oxide thin films were prepared by the sol-gel process, in which a precursor solution was prepared according to the literatures [17, 18]. In a typical synthesis method, 0.3 g V_2O_5 powder (99.5%, Sigma-Aldrich Co., Ltd.) was dissolved in 30 mL 30% H_2O_2 (35%, Chem-Supply Co., Ltd.) with rigorous stirring for 30 min. At this step, the reaction was strongly exothermic, and the solution's color changed from yellow to dark red sol upon a continuous stirring. After the two chemicals were mixed and aged for 2 days at room temperature, thin films were obtained by dip coating method using a dip coater (Model DC-150) performed at a dipping time of 2 min and a withdrawal rate of 6 mm/min on a glass slide substrate. The substrates were cleaned by means of ultrasonication for 30 min in deionized water, 20% acetone, 70% iso-propanol, and 20% ethanol, respectively. The as-deposited thin films were dried in ambient air for 24 h. The dried thin films were calcined at room temperature, 400, 450, and 500°C, in vacuum tube furnace at pressures of 2×10^{-2} and 2×10^{-3} mbar. To investigate an effect of heat treatment, calcination was conducted by 1- and 2-step calcination, in which each step employed a heating rate of 2°C/min and soaking time of 3 h. For the 1-step calcination, the thin films were heated in a tube furnace to 400, 450, and 500°C, soaked at this temperature for 3 h, and cooled down to room temperature. The 2-step calcination was carried out in a tube furnace at 500°C using the same heating and cooling cycles, noted that the glass slide substrate employed in this work began to soften at a temperature of ~500°C; thus, calcination temperature did not exceed 500°C. Process parameters for film deposition are provided in Table 1.

2.2. Thin Film Characterization. Crystal structure characterization of the thin films was performed by using an X-ray diffractometer (XRD; Rigaku TTRAX III). Morphology and thickness were studied by using a scanning electron microscope (SEM; Quanta 400 FEI) performed in a top and tilted view, respectively. Films' compositions, as well as an elemental mapping, were analyzed by using an energy dispersive X-ray spectrometer (EDS). Oxidation states of the elements were analyzed by using an X-ray photoelectron spectrophotometer (XPS; AXIS Ultra DLD). The binding energy was calibrated with carbon (C1s = 284.8 eV).

Electrical property of the thin film was determined by an AC four-point probe method. The measurement was conducted in a closed chamber with controlled temperature. During heating and cooling cycles, the film temperature was raised from room temperature to 100°C and subsequently reduced to room temperature step by step. The metal-to-insulator (MIT) phase transition temperature is defined as the average of the transition temperature (T_c) in the electrical property. The T_c is defined as follows [19]:

$$T_c = \frac{\left[T_c\,(\text{heating}) + T_c\,(\text{cooling})\right]}{2}. \tag{1}$$

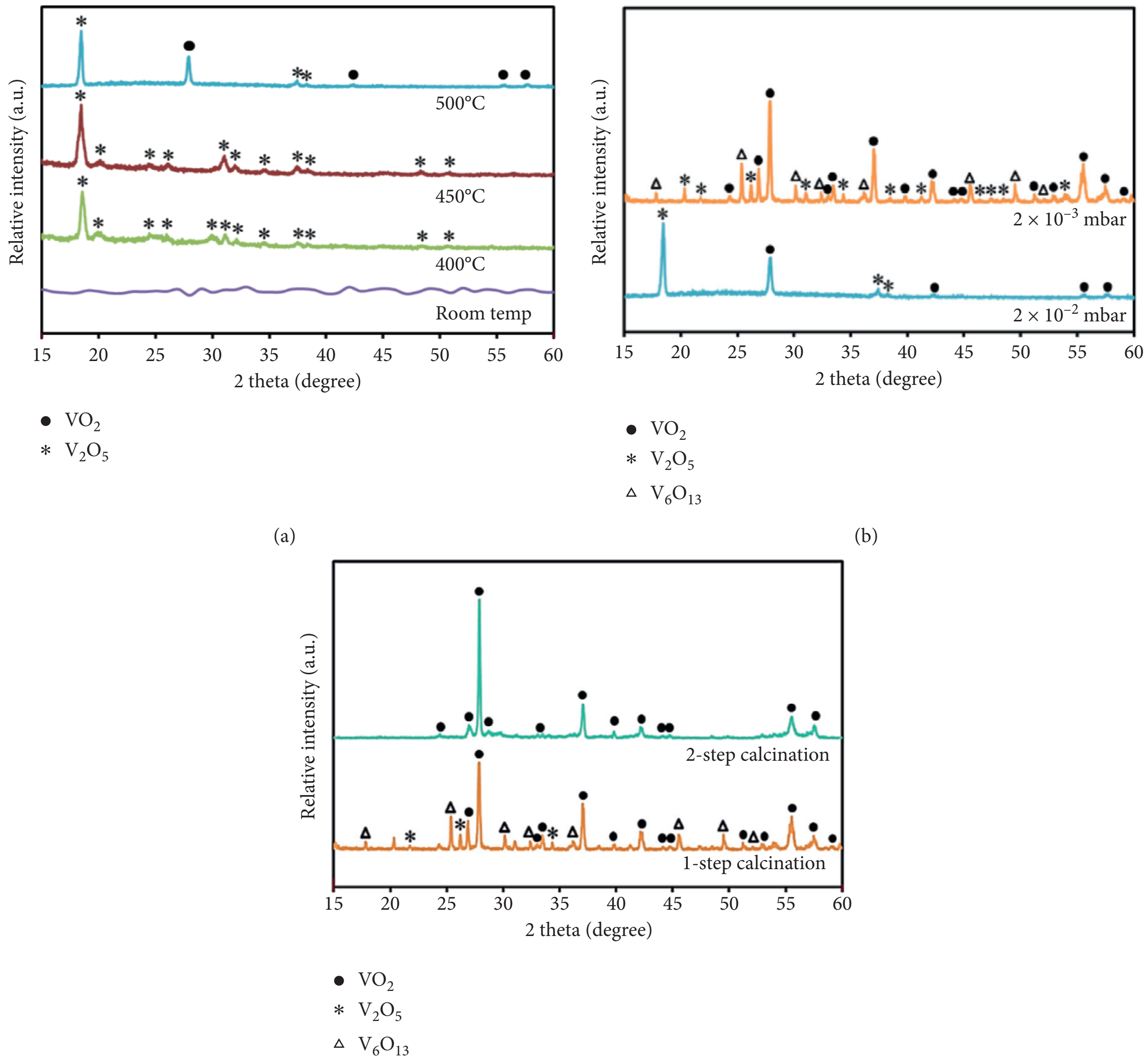
Transparency of the film coated on the soda lime glass was determined at 25 and 80°C by using a UV-Vis-NIR spectrophotometer (Shimadzu UV-3600 Plus) in a wavelength range 200–2500 nm. The bare soda lime glass was employed as a blank sample. Temperatures were controlled at 25 and 80°C with applied current electricity.

3. Results and Discussion

3.1. Synthesis of Vanadium Oxide Thin Films by Sol-Gel Dip Coating. It has been realized that it is difficult to fabricate pure monoclinic phase VO_2 thin film due to its instability under a general synthesis process. It is most likely that more stable vanadium oxides, such as V_2O_5, V_2O_3, and V_6O_{13}, are also present. These oxides block the reversible phase transition from metallic state to insulating state of the monoclinic VO_2. Among all of the V-O system, the V_2O_5 is a highly stable phase [20, 21]. Thus, in order to investigate the optimum synthesis condition, the presence of crystalline phases was initially considered. Figure 1 shows XRD

TABLE 1: Fabrication parameters of VO_2, V_2O_5, and V_6O_{13} thin films.

Phase	Furnace temperature (°C)	Vacuum pressure (mbar)	Calcination (step)
Amorphous	Room temperature	2×10^{-2}	1
V_2O_5	400	2×10^{-2}	1
V_2O_5	450	2×10^{-2}	1
VO_2+V_2O_5	500	2×10^{-2}	1
VO_2+V_2O_5+V_6O_{13}	500	2×10^{-3}	1
VO_2	500	2×10^{-3}	2



(a)

(b)

(c)

FIGURE 1: XRD patterns of the vanadium oxide thin films prepared at various conditions: (a) $T = $ RT, 400, 450, and 500°C ($P = 2\times10^{-2}$ mbar, 1-step calcination), (b) $P = 2\times10^{-2}$ and 2×10^{-3} mbar ($T = 500$°C, 1-step calcination), and (c) 1- and 2-step calcination ($P = 2\times10^{-3}$ mbar, $T = 500$°C).

patterns of the vanadium oxide thin films after calcined at various conditions summarized in Table 1.

For the 1-step calcination (Figure 1(a)), the temperatures were controlled at 400, 450, and 500°C, respectively, and the pressure was fixed at 2×10^{-2} mbar. XRD analysis of the dried thin film (denoted as "room temperature") revealed amorphous nature signified by the absence of diffraction peak. XRD patterns of the thin films calcined at 400 and 450°C show diffraction peaks at 18.7°, 20.1°, 24.7°, 26.2°, 30.1°, 31.3°, 32.4°, 34.8°, 37.6°, 38.6°, 48.6°, and 51.1°, which correspond to the (001), (101), (201), (110), (301), (400), (111), (211), (401), (311), (302), and (012) planes, respectively, of the orthorhombic V_2O_5 (ICCD card no. 04-015-2250). Increasing the calcination temperature results in an increased intensity of the XRD peaks, indicating an increase of film's crystallinity. For the thin film calcined at 500°C, its XRD pattern reveals the peaks at 27.7°, 42.5°, 55.7°, and 57.8° corresponding to the (011), (210), (220), and (211) planes, respectively, of the monoclinic VO_2 (ICCD card no. 00-043-1051). Diffraction peaks of the V_2O_5 at 18.7°, 37.6°, and 38.6°, which correspond to the (001), (401), and (311), planes, respectively, of the orthorhombic V_2O_5 (ICCD card no. 04-015-2250) are also observed. This result indicates a coexistence of the V_2O_5 and VO_2 phases by the 1-step calcination at 500°C. To further investigate the effect of calcination condition, the pressure of the vacuum tube furnace was varied to 2×10^{-3} mbar, while the temperature was maintained at 500°C. As shown in Figure 1(b), XRD patterns of the orthorhombic V_2O_5 are dramatically reduced while those of the monoclinic VO_2 are enhanced. In addition, diffraction peaks at 17.9°, 25.7°, 30.3°, 32.6°, 36.3°, 45.8°, 50.1°, and 52.3° corresponding to the (002), (110), (400), (−402), (−113), (005), (020), and (220) planes, respectively, of the V_6O_{13} phase (ICCD card no. 04-008-4854) are present. This result indicates that lower vacuum pressure, the less amount of oxygen, promoted the formation of the VO_2, as well as the V_6O_{13} minor phase. This reduction condition at a temperature above 400°C and a pressure below 2 Pa (2×10^{-2} mbar) which caused the conversion of V_2O_5 thin films to VO_2 has been reported by Ningyi et al. [22]. It was observed that the reduction process followed the sequence as $V_2O_5 \longrightarrow V_3O_7 \longrightarrow V_4O_9 \longrightarrow V_6O_{13} \longrightarrow VO_2$, namely, from V_nO_{2n+1} ($n = 2$–4, 6) to VO_2. Therefore, lower pressure favoured VO_2 formation caused by the oxygen deficiency [22, 23]. To further investigate the effect of calcination time, the calcination soaking time was increased from 3 to 24 h. It was found that the thin film consisted of only V_2O_3 phase.

Figure 1(c) shows XRD patterns of the thin films prepared by means of 1-step and 2-step calcination in the vacuum tube furnace at 500°C with the pressure of 2×10^{-3} mbar. It is evident that only diffraction peaks of the monoclinic VO_2 are present in the film prepared by 2-step calcination caused by the reduction process of V_2O_5 and V_6O_{13}.

SEM surface analysis of the VO_2 thin film prepared by the 2-step calcination process at 500°C with the pressure of 2×10^{-3} mbar (Figures 2(a) and 2(b)) reveals very fine VO_2 grains with irregular shape of ~220–380 nm in size surrounded by an amorphous phase of anhydrous vanadium oxide from gelation [24]. Film's thickness determined from a cross-sectional image (Figure 2(c)) is approximately 69–74 nm. Elemental composition analyzed by EDS (Figure 2(d)) reveals that the sample comprises of Si, O, Na, Ca, V, Mg, and Al at the amount of 30.1, 51.0, 8.5, 5.7, 1.5, 2.6, and 0.6 At.%, respectively. The V and O correspond to the VO_2 thin film, and the others (and part of O) correspond to the glass slide substrate. The elemental mappings of Si from the glass substrate and V from the VO_2 film (Figures 2(e) and 2(f)) reveal homogeneous distribution of the VO_2 grains in the thin film.

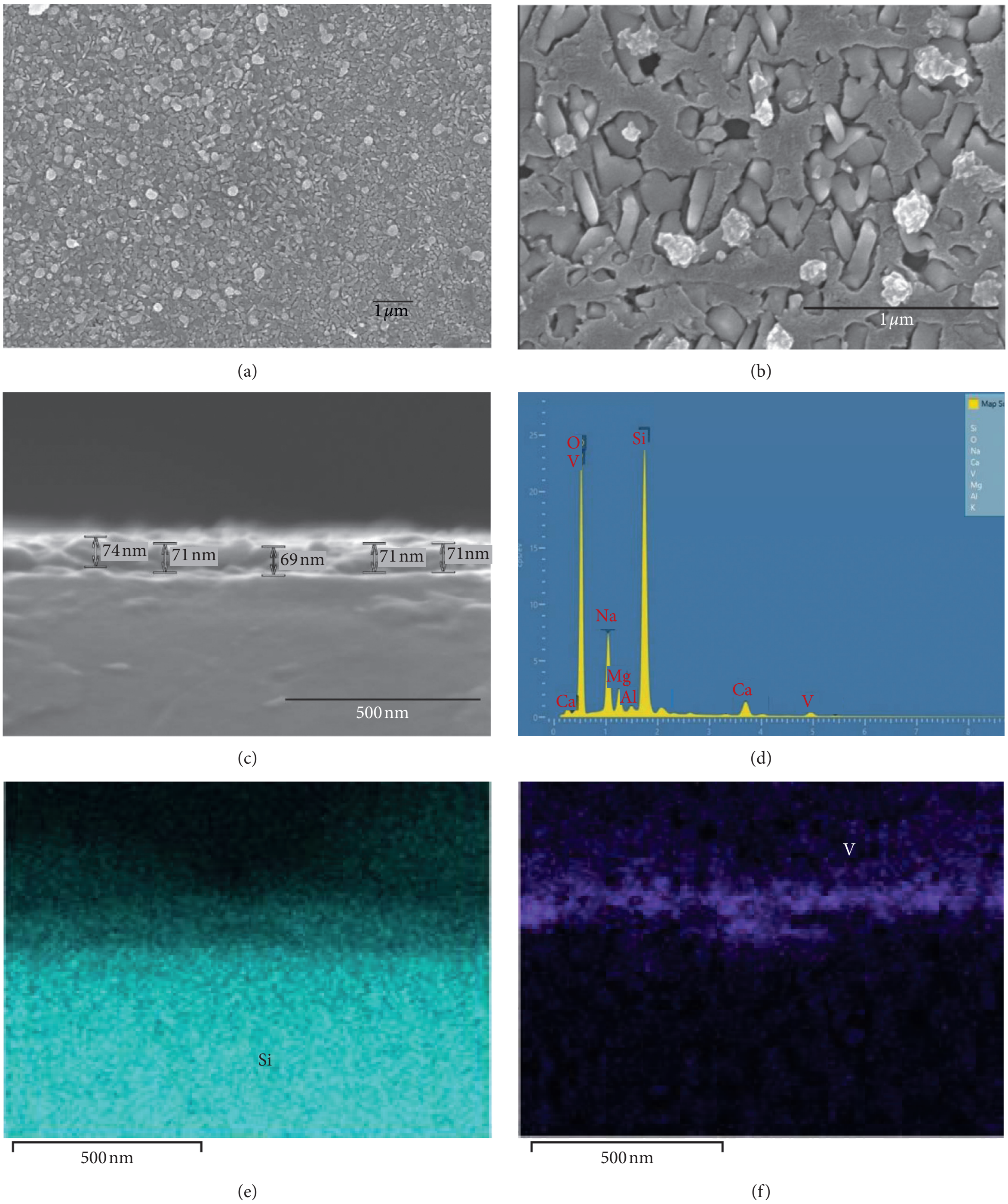
Typical surface XPS spectra of the VO_2 sample are depicted in Figure 3. Detail-scan spectra were employed to investigate the valence electron of the V and O and to confirm phase purity of the thin films. According to the standard binding energy, a typical two-peak structure (2p3/2 and 2p1/2) due to the spin-orbit splitting was observed [19]. The peaks at 516.1 and 523.8 eV shown in Figure 3(a) correspond to binding energies for $V^{4+}_{2p3/2}$ and $V^{4+}_{2p1/2}$, respectively. The peaks at 517.6 eV and 525.2 eV correspond to $V^{5+}_{2p3/2}$ and $V^{5+}_{2p1/2}$, respectively. This result indicated atmospheric oxidation of a small amount of V^{5+} at the surface of the sample. The binding energy of O_{1s} appears at 530.0, 531.8, and 532.8 eV, which correspond to the V-O bonding, O--H bonding, and part of physically adsorbed H_2O, respectively, as shown in Figure 3(b). Besides these major energy levels, a number of less intense satellite peaks are also present. These V_{2p} satellite peaks are attributed to the V_2O_3. The XPS composition analysis result confirms the presence of VO_2, as well as a small amount of V_2O_5 and V_2O_3 [25, 26].

3.2. Electrical Properties of VO_2 (M) Thin Films.

Figure 4(a) illustrates the temperature dependence of electrical resistance of the VO_2 thin films on the soda lime glass prepared by the 2-step calcination process at 500°C with the pressure of 2×10^{-3} mbar. The hysteresis loop indicates the metal-to-insulator transformation and the quality of the thin film. It was observed that surface resistance of the thin film decreased upon the heating process and increased upon the cooling process. The transition temperature, determined according to equation (1), was approximately 68°C (T_c = (80°C + 56°C)/2). Furthermore, resistivity profile of the VO_2 thin films was nearly flat above the room temperature (semiconductor phase), indicating that the transition occurs at 68°C (metal-insulator transition phase) and constant at 80°C (metal phase), respectively [7, 27]. The VO_2 thin films were transformed from monoclinic to tetragonal (rutile) phase at the transition temperature.

3.3. Optical Properties of VO_2 (M) Thin Films.

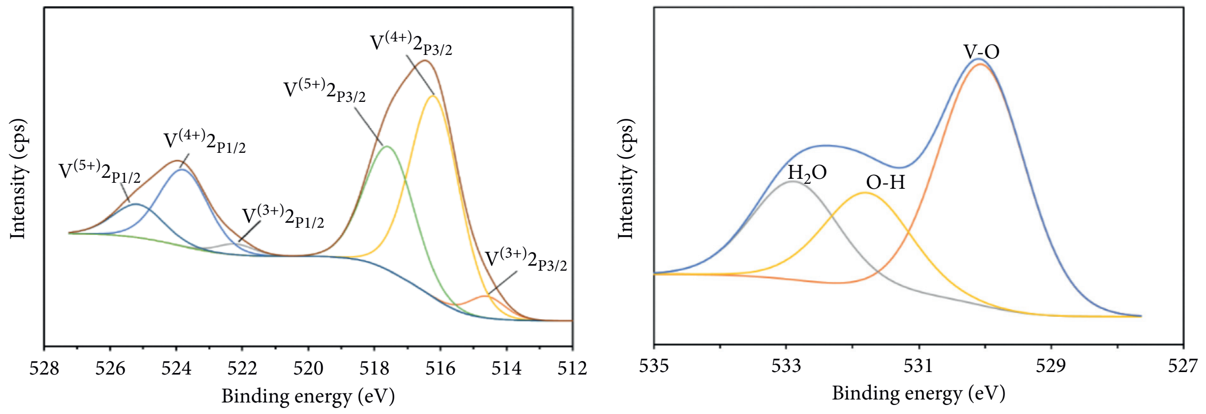
Figure 4(b) illustrates the optical transmittance spectra measured at 25 and 80°C of the VO_2 thin films. Visible optical transmittance remains at ~40–50% when the sample's temperature changes from 25 to 80°C. However, for a near infrared (NIR) region wavelength range of 800–2500 nm, the optical transmittance measured at 25°C is clearly



(a) (b) (c) (d) (e) (f)

Figure 2: SEM images of the surfaceVO2 thin films at a resolution of x10,000 (a) and x50,000 (b) and cross-sectional image at x100,000 (c). Film's elemental composition analyzed by EDS (d), Si mapping (e), and V mapping (f).

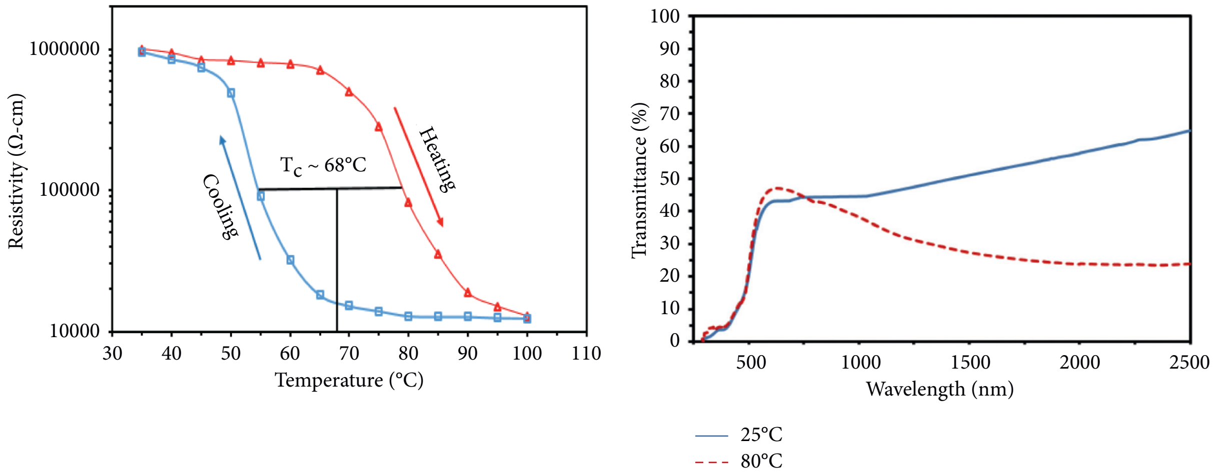
higher than that measured at 80°C. Moreover, the optical transmittance increases with increasing wavelength at 25°C, while it reduces with increasing wavelength at 80°C, indicating phase transition when the temperature is raised from 25 to 80°C. Thus, the thin film exhibits thermochromic properties [28–30].



(a)

(b)

Figure 3: XPS spectra of the VO_2 thin film; curve-fitted V_{2p} (a) and O_{1s} (b) core level spectra.



(a)

(b)

Figure 4: Thermal hysteresis loop (a) and the optical transmittance spectra (b) of VO_2 thin films.

4. Conclusions

A simple and effective synthesis method for the preparation of the vanadium dioxide thin films on the glass slide substrate has been demonstrated by a sol-gel dip coating at low temperature of 500°C in a vacuum tube furnace at a pressure of 2×10^{-3} mbar. The calcination was performed by 2 steps, in which thin films were heated in a tube furnace to 500°C at a heating rate of 2°C/min, soaked for 3 h, and cooled down to room temperature, and the same process was repeated. The analyses revealed that the thin films consisted of monoclinic VO_2 phase and a small amount of V_2O_5 and V_2O_3 phases. The very fine VO_2 grains with irregular shape of ~220–380 nm in size were homogeneously distributed on the surface. The film's thickness was in a range of 69–74 nm. The monoclinic to tetragonal (rutile) phase transition temperature was 68°C. The thin films exhibited good thermochromic properties. The low-temperature synthesis would be beneficial for thermochromic thin film coating on a conventional soda lime glass panel. In addition, as no intermediate gas was employed during calcination, the process would be suitable for large-scale fabrication.

Data Availability

The data used to support the finding of this study are included within the article.

Conflicts of Interest

The authors declare that there are no conflicts of interest regarding the publication of this paper.

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